CERTIFICATE

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Document Name: HACH 8008: 1, 10--Phenanthroline Method Using FerroVer Iron Reagent for Water

CFR Section(s): 40 CFR 136.3(a)

Standards Body: Hach Chemical Company

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Iron, Total

DOC316.53.01053

USEPA[1] FerroVer® Method[2] **Method 8008**

0.02 to 3.00 mg/L **Powder Pillows or AccuVac® Ampuls**

Scope and Application: For water, wastewater and seawater; digestion is required for determining total iron

[1] USEPA approved for reporting wastewater analysis, *Federal Register,* June 27, 1980; 45 (126:43459)

[2] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Cell orientation	Sample cell	Adapter
DR 5000	2495402	Fill line faces user	2427606	—
DR 3900	2495402	Fill line faces user	2427606	LZV846 (A)
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	2122800	LZV584 (C)

Before starting the test:

Digestion is required for determining total iron for EPA reporting purposes. Use the mild or vigorous digestion. Refer to the *Water Analysis Guide* for more information.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust. See the user manual for more information.
Adjust pH of stored samples before analysis.
For turbid samples, treat the blank with one 0.1-g scoop of RoVer Rust Remover. Swirl to mix.

Collect the following items:

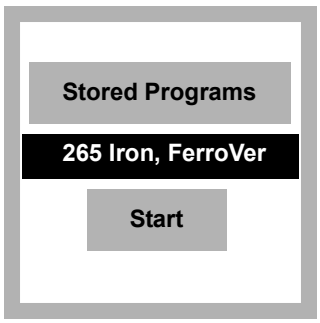
Description	Quantity
Powder Pillow Test:	
FerroVer® Iron Reagent Powder Pillow	1
Sample Cells (see *Instrument-specific information*)	2

Collect the following items: (continued)

Description	Quantity
AccuVac® Ampul test:	
FerroVer® Iron Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample Cells (see *Instrument-specific information*)	1
Stopper of 18 mm tubes	1

See *Consumables and replacement items* for reorder information.

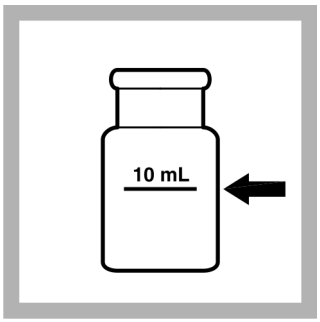
FerroVer method for powder pillows



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

Refer to the user manual for orientation.



2. Prepared sample: Fill a clean sample cell with 10 mL of sample

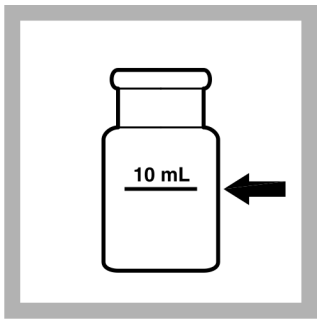
3. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell. Swirl to mix.

Accuracy is not affected by undissolved powder.

4. Start the instrument timer.

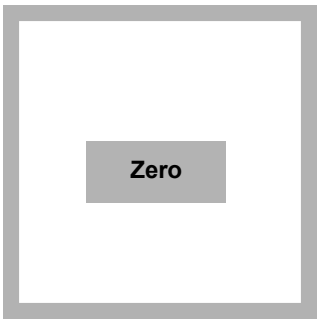
A three-minute reaction period will begin. An orange color will form, if iron is present.

(Allow samples that contain rust to react for at least 5 minutes.)



5. Blank preparation: Fill a second sample cell with 10 mL of sample.

6. When the timer expires, insert the blank into the cell holder.



7. ZERO the instrument.

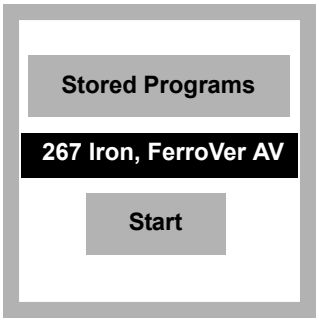
The display will show:

0.00 mg/L Fe

8. Insert the prepared sample into the cell holder.

READ the results in mg/L Fe.

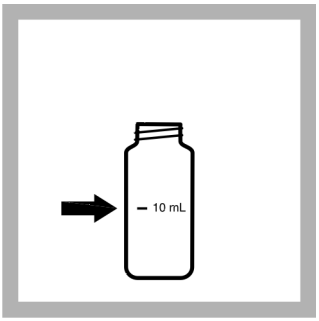
FerroVer method for AccuVac® Ampuls



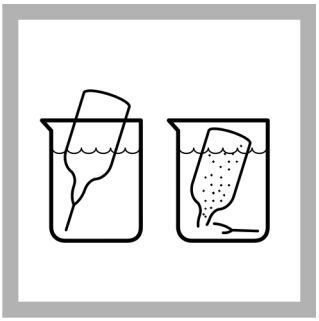
1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

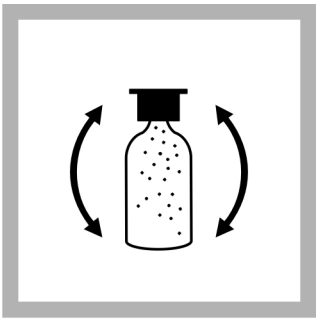
Refer to the user manual for orientation.



2. Blank Preparation: Fill a round sample cell with 10 mL of sample.



3. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker. Fill a FerroVer Iron AccuVac® Ampul with sample from the beaker. Keep the tip immersed while the Ampul fills completely.



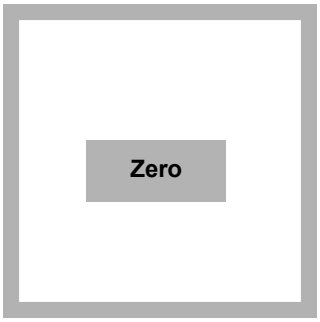
4. Quickly invert the Ampul several times to mix.

Accuracy is not affected by undissolved powder.

5. Start the instrument timer.

A three-minute reaction period will begin. An orange color will develop if iron is present.

6. Wipe the blank and insert it into the cell holder.



7. ZERO the instrument.

The display will show:

0.00 mg/L Fe

8. Wipe the Ampul and insert it into the cell holder.

READ the results in mg/L Fe.

Interferences

Table 2 Interfering substances

Interfering substance	Interference level
Calcium, Ca^{2+}	No effect at less than 10,000 mg/L as $CaCO_3$.
Chloride, Cl^-	No effect at less than 185,000 mg/L.
Copper, Cu^{2+}	No effect. Masking agent is contained in FerroVer Reagent.
High Iron Levels	Inhibit color development. Dilute sample and re-test to verify results.
Iron Oxide	Requires mild, vigorous or Digesdahl digestion. After digestion, adjust sample to pH 3–5 with sodium hydroxide, then analyze.
Magnesium	No effect at 100,000 mg/L as calcium carbonate.

Table 2 Interfering substances (continued)

Interfering substance	Interference level
Molybdate Molybdenum	No effect at 50 mg/L as Mo.
High Sulfide Levels, S^{2-}	**1.** Treat in fume hood or well-ventilated area. Add 5 mL hydrochloric acid[1], ACS to 100 mL sample in a 250 mL Erlenmeyer flask. Boil 20 minutes. **2.** Cool. Adjust pH to 3–5 with Sodium Hydroxide[1]. Readjust volume to 100 mL with deionized water. **3.** Analyze using *FerroVer method for powder pillows* or *FerroVer method for AccuVac® Ampuls*.
Turbidity	**1.** Add 0.1 g scoop of RoVer® Rust Remover to the blank. Swirl to mix. **2.** Zero the instrument with this blank. **3.** If sample remains turbid, add three 0.2 g scoops of RoVer to a 75 mL sample. Let stand 5 minutes. **4.** Filter through a Glass Membrane Filter and Filter Holder[1]. **5.** Use the filtered sample as the prepared sample and the blank.
Extreme Sample pH	Adjust pH to 3–5.
Highly Buffered Samples	Adjust pH to 3–5.

[1] See *Optional reagents and apparatus*.

Sample collection, preservation and storage

- Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately.
- To preserve samples, adjust the pH to 2 or less with concentrated nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature.
- Before analysis, adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution.
- Correct the test result for volume additions.
- If only dissolved iron is to be determined, filter the sample before acid addition.

Accuracy check

Standard additions method (sample spike)
Required for accuracy check:

- Iron Voluette® Ampule Standard, 25 mg/L
- Ampule breaker
- TenSette Pipet and Pipet Tips

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select Options>More>Standard Additions from the instrument menu.
3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open the standard solution ampule.
5. Prepare a 0.1 mL sample spike by adding 0.1 mL of standard to 10 mL of unspiked sample. Start the instrument timer. After the timer expires, read the result.

6. Prepare a 0.2 mL sample spike by adding 0.1 mL of standard to the 0.1 mL sample spike. Start the instrument timer. After the timer expires, read the result.
7. Prepare a 0.3 mL sample spike by adding 0.1 mL of standard to the 0.2 mL sample spike. Start the instrument timer. After the timer expires, read the result.
8. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard additions method for AccuVac Ampuls (sample spike)

1. Fill three mixing cylinders each with 50 mL of sample and spike with 0.2 mL, 0.4 mL and 0.6 mL of standard. Stopper and invert to mix.
2. Transfer 40 mL from each of the three mixing cylinders to three 50 mL beakers.
3. Analyze each standard addition sample as described in the *FerroVer method for AccuVac® Ampuls*.
4. Accept each standard additions reading. Each addition should reflect approximately 100% recovery.

Standard solution method

Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- Iron Standard Solution, 100 mg/L
- 100-mL volumetric flask
- Class A volumetric pipet, 2 mL
- Deionized water
- Pipet filler

1. Prepare a 2.00-mg/L Fe standard solution as follows:
   a. Pipet 2.00 mL of Iron Standard Solution, 100 mg/L, into a 100 mL volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the 2.00 mg/L Fe standard solution in place of the sample. Follow the *FerroVer method for powder pillows* test procedure.
3. To adjust the calibration curve using the reading obtained with the Standard Solution, select Options>More>Standard Adjust from the instrument menu.
4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value. Mixed-parameter standards are also available to simulate various matrices.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
265	2.00 mg/L Fe	1.99–2.01 mg/L Fe	0.021 mg/L Fe
267	2.00 mg/L Fe	1.98–2.02 mg/L Fe	0.023 mg/L Fe

Summary of method

FerroVer Iron Reagent converts all soluble iron and most insoluble forms of iron in the sample to soluble ferrous iron. The ferrous iron reacts with the 1-10 phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration. Test results are measured at 510 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
FerroVer® Iron Reagent Powder Pillows (for 10-mL sample)	1	100/pkg	2105769
OR			
FerroVer® Iron Reagent AccuVac® Ampuls	1	25/pkg	2507025

Required apparatus

Description	Quantity	Unit	Catalog number
Beaker, 50 mL	1	each	50041H
Sample cell, 10 mL round, 25 x 54 mm	1	each	2122800
Sample cell, 10 mL round, 25 x 60 mm	1	6/pkg	2427606
Sample cell, 10 mL square, matched pair	2	2/pkg	2495402
Stopper for 18 mm tube	1	6/pkg	173106

Recommended standards

Description	Unit	Catalog number
Iron Standard Solution, 100 mg/L	100 mL	1417542
Iron Standard Solution, 10 mL Voluette® Ampule, 25 mg/L as Fe	16/pkg	1425310
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	2833749
Metals Drinking Water Standard, HR for Cu, Fe, Mn	500 mL	2833649
Water, deionized	4 L	27256
Pipet, TenSette, 0.1–1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001	1000/pkg	2185628
Flask, volumetric, Class A, 100 mL	each	1457442
Pipet, volumetric, Class A, 2.00 mL	each	1451536
Pipet Filler, safety bulb	each	1465100

Optional reagents and apparatus

Description	Unit	Catalog number
Beaker, 50 mL	each	50041H
Cylinder, mixing, 50 mL	each	189641
Hydrochloric Acid, concentrated	500 mL	13449
Nitric Acid, concentrated	500 mL	15249

Optional reagents and apparatus (continued)

Description	Unit	Catalog number
Sodium Hydroxide Standard Solution, 5.0 N	100 mL	245032
Glass Membrane Filter, 47 mm	100/pkg	253000
Glass Membrane Filter Holder	each	234000
RoVer Rust Remover	454 g	30001
Spoon, measuring, 0.1 g	each	51100

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